



# Surface Diagnostics in Tribology Technology and Advanced Coatings Development

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# Surface Diagnostics in Tribology Technology and Advanced Coatings Development

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## Abstract

This paper discusses the methodologies used for surface property measurement of thin films and coatings, lubricants, and materials in the field of tribology. Surface diagnostic techniques include scanning electron microscopy, transmission electron microscopy, atomic force microscopy, stylus profilometry, x-ray diffraction, electron diffraction, Raman spectroscopy, Rutherford backscattering, elastic recoil spectroscopy, and tribology examination. Each diagnostic technique provides unique information. It should be possible to coordinate the different pieces of information provided by these diagnostic techniques into a coherent self-consistent description of the surface properties. Examples are given on the nature and character of thin diamond films.

Keywords: measurement, surface methodology, diamond, films and coatings, and tribology

## 1.0 Introduction

Several diagnostic techniques are now available for measuring the properties and composition of any solid surface. Because the surface plays a crucial role in many mechanical, physical, and chemical processes, such as adhesion, friction, wear, oxidation, and corrosion, these diagnostic techniques have established their importance in a number of scientific, industrial, and commercial fields [1].

Tribology is defined as the science and technology of interacting surfaces in relative motion and of related subjects and practices, such as adhesion, friction, lubrication, and wear [2]. It is now believed that proper attention to tribology, especially surface engineering and technology, research, application, and education, could lead to economic savings of between 1.3 and 1.6% of the gross national product (GNP). Tribology impacts strongly on the national economy and on the lifestyles of most people. Wear shortens product life and friction results in wasted energy consumption. As material and energy shortages develop, greater demand will arise for longer product life, increased wear resistance, and reduced energy consumption through lubrication and surface coatings. During the last decade significant progress has taken place in the development of advanced surface films and coatings used in tribology technology. Their number is growing; some of the most exciting recent developments are superhard coatings and films, such as chemical-vapor-deposited (CVD) diamond, diamondlike carbon (DLC), and cubic boron nitride (c-BN) films [3].

This paper deals with the application of surface diagnostic techniques in the field of tribology and in the development of advanced tribological coatings, particularly diamond films and related coatings. These techniques can probe complex surfaces of thin films and coatings, lubricants, and materials and clarify their interactions in mechanical systems and processes. Examples are given on the nature and character of microwave-plasma-assisted CVD diamond films. Some earlier data and experimental details on this research are given in the references [4-6].

## 2.0 CVD Diamond

A variety of techniques can be used to characterize CVD diamond films: scanning electron microscopy and transmission electron microscopy (SEM and TEM), to determine surface morphology, micro-structure, and grain size; surface profilometry and atomic force microscopy (AFM), to measure surface roughness and to determine surface morphology; Rutherford backscattering (RBS) and elastic recoil spectroscopy (ERS), to determine the composition (including hydrogen); Raman spectroscopy, to characterize the atomic bonding state and quality of diamond; x-ray diffraction (XRD), to determine the crystal orientation of diamond; and tribological examination, to determine the coefficient of friction, the friction behavior, and the wear rate.

It is generally accepted [7] that for a material to be recognized as diamond it must have all of the following characteristics:

1. Crystalline diamond morphology and micro-structure visible by electron microscopy
2. Single-phase diamond crystalline structure detectable by x-ray or electron diffraction
3. Clear, sharp diamond peak at  $1332\text{ cm}^{-1}$  in a Raman spectrum
4. The carbon element
5. Low equilibrium coefficient of friction (0.01 to 0.05) in air

The following case studies focus attention primarily on microwave-plasma-assisted CVD diamond films.

## 2.1 Electron Microscopy, Stylus Profilometer, and Atomic Force Microscopy

Transmission electron microscopy offers two methods of specimen observation: image mode and diffraction mode [8]. In image mode, analysis of transmitted electron images yields information both about atomic structure and about defects present in the material. In diffraction mode, an electron diffraction pattern is obtained from the specimen area illuminated by the electron beam. The electron diffraction pattern is entirely equivalent to an x-ray diffraction pattern.

Scanning electron microscopy (with energy dispersive spectroscopy) is the most useful tool when the researcher needs not only morphological and topographical information about surfaces but also information concerning the composition of near-surface regions of the material. Although diamond is an insulator, it can be studied by using low primary electron beam voltages (5 keV or less) if one is willing to compromise image resolution to some extent. If the diamond is coated with a thin conducting film (10 to 20 nm thick) of carbon, gold, or some other metal,

the coated diamond can be studied with an image resolution of 1 to 50 nm.

The stylus profilometer (mechanical profilometer) is today commonly used for measuring surface roughness and analyzing topography. A diamond stylus with a tip radius of a few micrometers moves up and down as it dragged across a specimen surface. This up-and-down motion effectively replicates the surface topography.

The grain size, surface morphology, and surface roughness of a microwave-plasma-assisted CVD diamond film can be controlled by varying the deposition parameters, such as gas-phase chemistry parameters and temperatures (e.g., Table 1 and [5]). The grain size and surface roughness data were obtained by using TEM and stylus profilometry. The CVD diamond films referred to in Table 1 can be divided into three groups by grain size: fine, medium, and coarse grain. The grain sizes of the fine-grain diamond films were determined from bright- and dark-field electron photomicrographs to be between 20 and 100 nm. The medium- and coarse-grain diamond films have grain sizes estimated at 1000 to 1500 nm and 3300 nm, respectively. The average surface roughness of the diamond films measured by a surface profilometer increases as the grain size increases, as shown in Fig. 1. Figure 2 shows scanning electron photomicrographs of fine-, medium-, and coarse-grain diamond films. Triangular crystalline facets typical of diamond are clearly evident on the surfaces of the medium- and coarse-grain films.

In an atomic force microscope, to obtain topographical information, a probe tip traverses across a diamond surface and senses the force of interaction between itself and the diamond surface. By monitoring the tip deflected necessary to maintain a constant interacting force surface topographical data can be obtained on a nanometer scale. Figure 3 is an AFM image of a fine-grain CVD diamond film. The surface has a granulated or spherulitic morphology (i.e., it contains spherical asperities of different sizes).

TABLE 1.—DEPOSITION CONDITIONS FOR DIAMOND FILMS OF VARIOUS GRAIN SIZES

Condition	Substrate <sup>a</sup>						
	Si (100)	Si (100)	$\alpha$ -SiC	$\alpha$ -SiC	$\alpha$ -SiC	$\text{Si}_3\text{N}_4$	$\text{Si}_3\text{N}_4$
Deposition temperature, °C	860±20	1015±50	1015±50	965±50	860±20	965±50	860±20
Gaseous flow rate, cm <sup>3</sup> /min:							
CH <sub>4</sub>	4	3.5	3.5	3.5	4	3.5	4
H <sub>2</sub>	395	500	500	500	395	5800	395
O <sub>2</sub>	1	0	0	0	1	0	1
Pressure, torr	5	40	40	40	5	40	5
Microwave power, W	500	100	1000	1000	500	1000	1500
Deposition time, hr	10.5	140	14	22	21	22	521
Thickness, nm	1000	4200	5000	8000	1000	7000	800
Grain size, nm	20-100	1100	3300	1500	22-100	1000	22-100
Surface roughness rms, nm	15	63	160	92	50	52	35

<sup>a</sup>Scratched with 0.5-mm diamond paste.

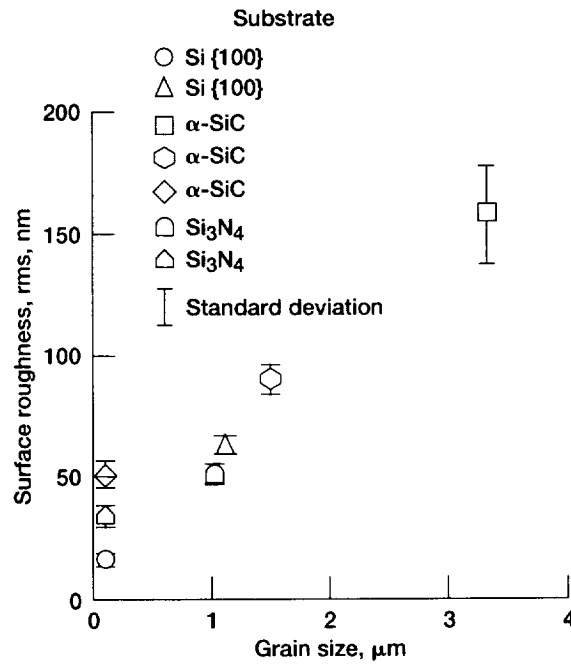


Figure 1.—Surface roughness as function of grain size for diamond films.

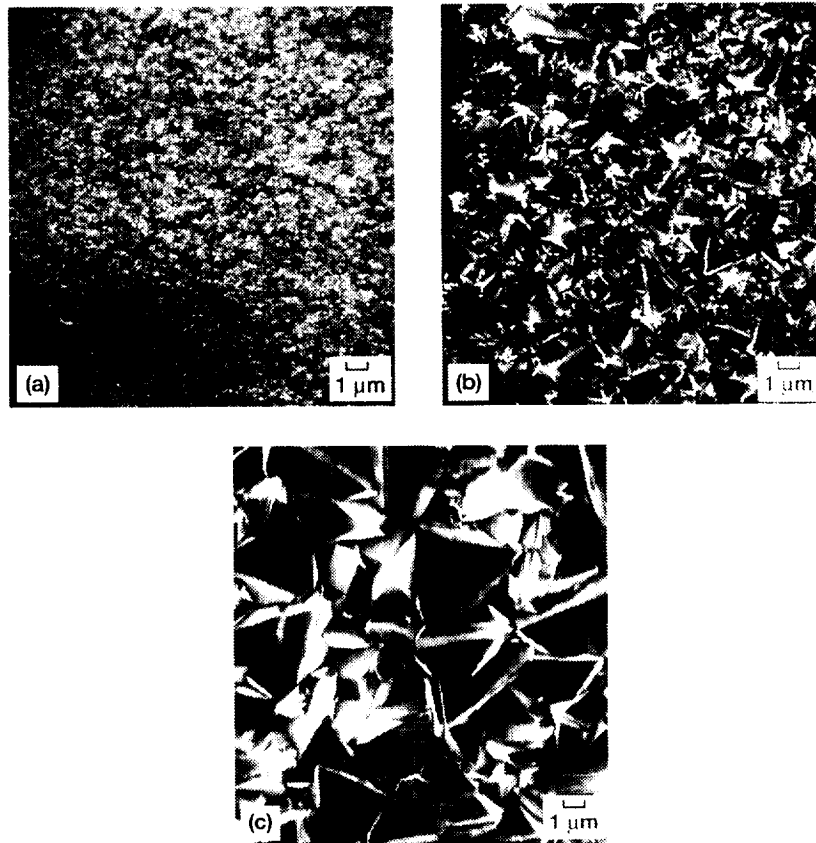


Figure 2.—Scanning electron micrographs of diamond films. (a) Fine-grain (20 to 100 nm) diamond film on {100} silicon substrate; rms surface roughness, 15 nm. (b) Medium-grain (1100 nm) diamond film on {100} silicon substrate; rms surface roughness, 63 nm. (c) Coarse-grain (3300 nm) diamond film on {100} α-silicon carbide substrate; rms surface roughness, 160 nm.

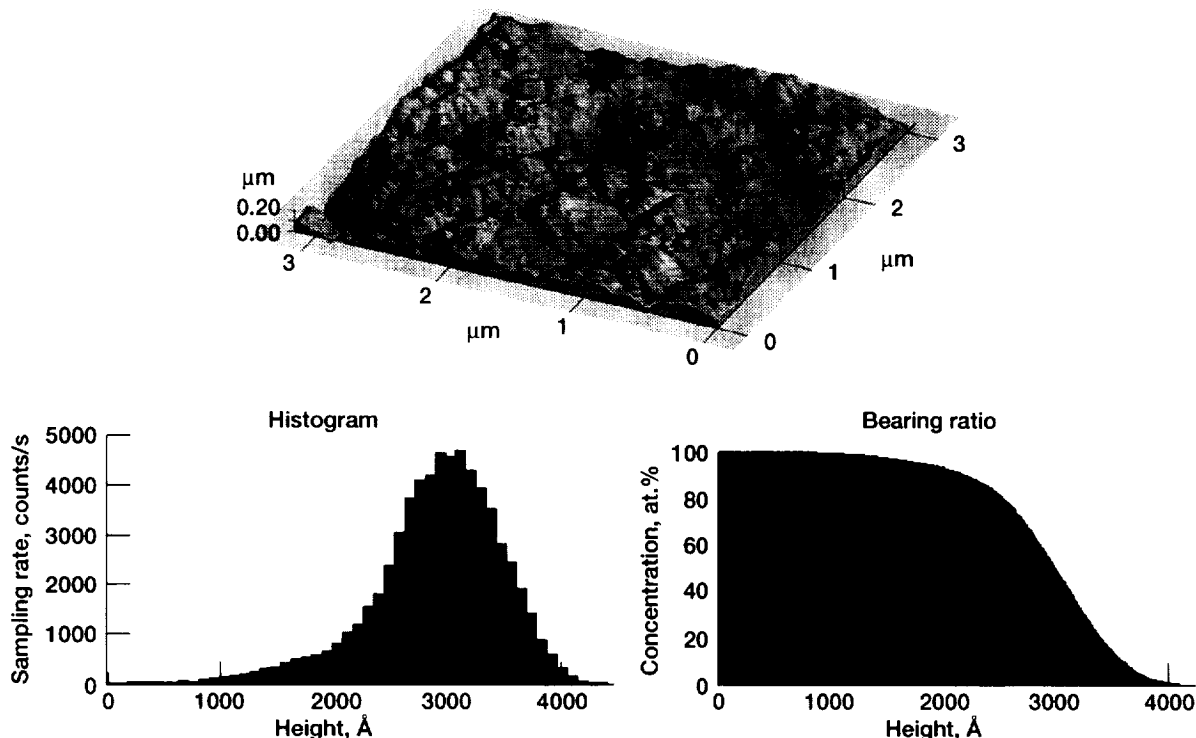


Figure 3.—Atomic force micrograph of chemical-vapor-deposited, fine-grain diamond film on mirror-polished silicon substrate.

## 2.2 X-Ray Diffraction and Electron Diffraction

X-ray diffraction is a powerful technique used to uniquely identify the crystalline phases present in materials and to measure the structural properties of these phases [8]. Although x-ray diffraction is not inherently a surface diagnostic technique, it offers unparalleled accuracy in the measurement of atomic spacing.

XRD was used to determine the structure and crystal orientation of the CVD diamond films. Typical x-ray diffraction patterns for the fine- and medium-grain diamond films (Fig. 4 [5]) show peaks representing only the diamond film and the silicon substrate. Diffraction peaks corresponding to the  $\{111\}$ ,  $\{220\}$ ,  $\{311\}$ , and  $\{400\}$  planes, reflective of diamond, are clearly evident. The intensity ratios  $I\{220\}/I\{111\}$  were calculated from the x-ray diffraction patterns for these films and found to be 1.3 and 0.04, respectively. The powder diffraction pattern of diamond with random crystal orientation (ASTM 6-0675) gives  $I\{220\}/I\{111\} = 0.27$ . Thus, most of the crystallites in the fine-grain diamond film are oriented along the  $\langle 110 \rangle$  direction, whereas most of the crystallites in the medium-grain diamond films are oriented along the  $\langle 111 \rangle$  direction. The well-formed triangular facets observed in SEM photomicrographs of medium- and coarse-grain diamond films confirm the  $\langle 111 \rangle$  crystal orientation.

Figure 5 presents a TEM selected-area diffraction pattern (SAD), a TEM bright-field photomicrograph, and a TEM dark-field photomicrograph of a free-standing, fine-grain CVD diamond film [4].

Diffraction rings and dots can be observed in Fig. 5(a). The d spacings of the diffraction rings were calculated by using an aluminum SAD as a calibration standard and were found to match well with the known diamond d spacings. No evidence of nondiamond carbon was found in the SAD. This observation indicates that the nondiamond concentration in the diamond film was extremely small.

Careful observation of Fig. 5(b) revealed various nuclei-like regions marked N. Diamond grains are distributed radially outward from these nuclei. A grain boundary is formed where the grains from various nuclei meet. As previously mentioned, the grain sizes of the fine-grain CVD diamond films estimated from the bright- and dark-field photomicrographs varied from 20 to 100 nm.

## 2.3 Raman Spectroscopy

Raman spectroscopy is primarily a structural characterization tool [8]. Raman spectra are more sensitive to the lengths, strengths, and arrangement of bonds in a material than to chemical composition. Raman spectra of crystals likewise reflect the details of defects and disorder than to trace impurities and related chemical imperfections. The laser-optical Raman technique can determine with great confidence the atomic bonding states of the carbon atoms ( $sp^2$  for graphite or  $sp^3$  for diamond) due to their different vibrational modes [9]. Raman spectra result from the inelastic scattering of optical photons by lattice vibration phonons.

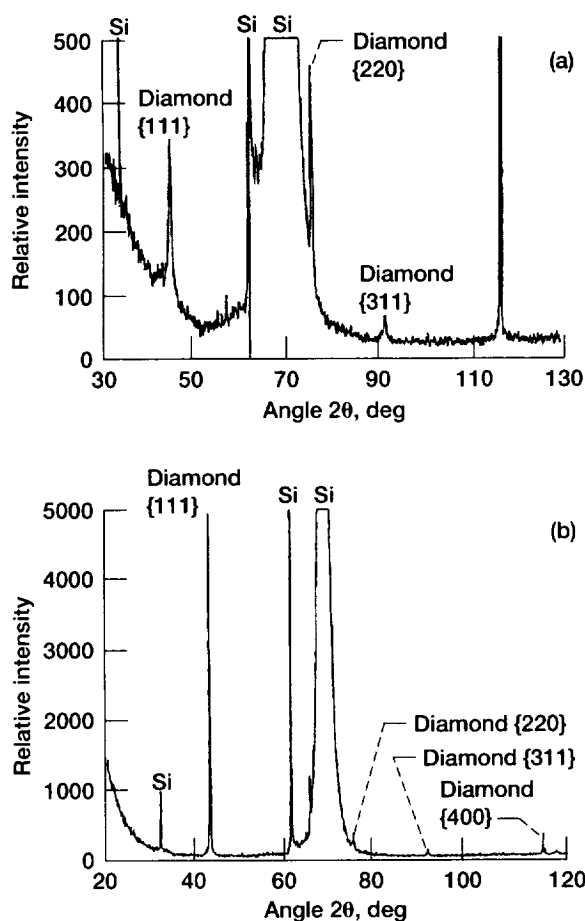


Figure 4.—X-ray diffraction patterns of diamond films. (a) Fine-grain (20 to 100 nm) diamond film on {100} silicon substrate. (b) Medium-grain (1100 nm) diamond film on {100} silicon substrate.

Typical Raman spectra of the fine- and medium-grain diamond films (Fig. 6) show one Raman band centered at  $1332\text{ cm}^{-1}$  and one centered around  $1530\text{ cm}^{-1}$ . The sharp peak at  $1332\text{ cm}^{-1}$  is characteristic of the  $\text{sp}^3$  bonding of the diamond form of carbon in the film. The very broad peak centered around  $1530\text{ cm}^{-1}$  is attributed to the  $\text{sp}^2$  bonding of the nondiamond forms of carbon (graphite and other carbon) [10–12].

More diamond is produced in larger grained CVD diamond films (e.g., Fig. 6) than in fine-grain films, as is evident from the relative intensities of the diamond and nondiamond carbon Raman bands [5]. However, the ratio of the intensities of the Raman responses at  $1332$  and centered around  $1530\text{ cm}^{-1}$  does not indicate the ratio of diamond to nondiamond carbon present in a particular film, since the Raman technique is approximately 50 times more sensitive to  $\text{sp}^2$ -bonded (nondiamond) carbon than to  $\text{sp}^3$ -bonded (diamond) carbon [11]. Thus, the peak centered around  $1530\text{ cm}^{-1}$  for each film represents a much smaller amount of nondiamond carbon in these diamond films than appears at first glance.

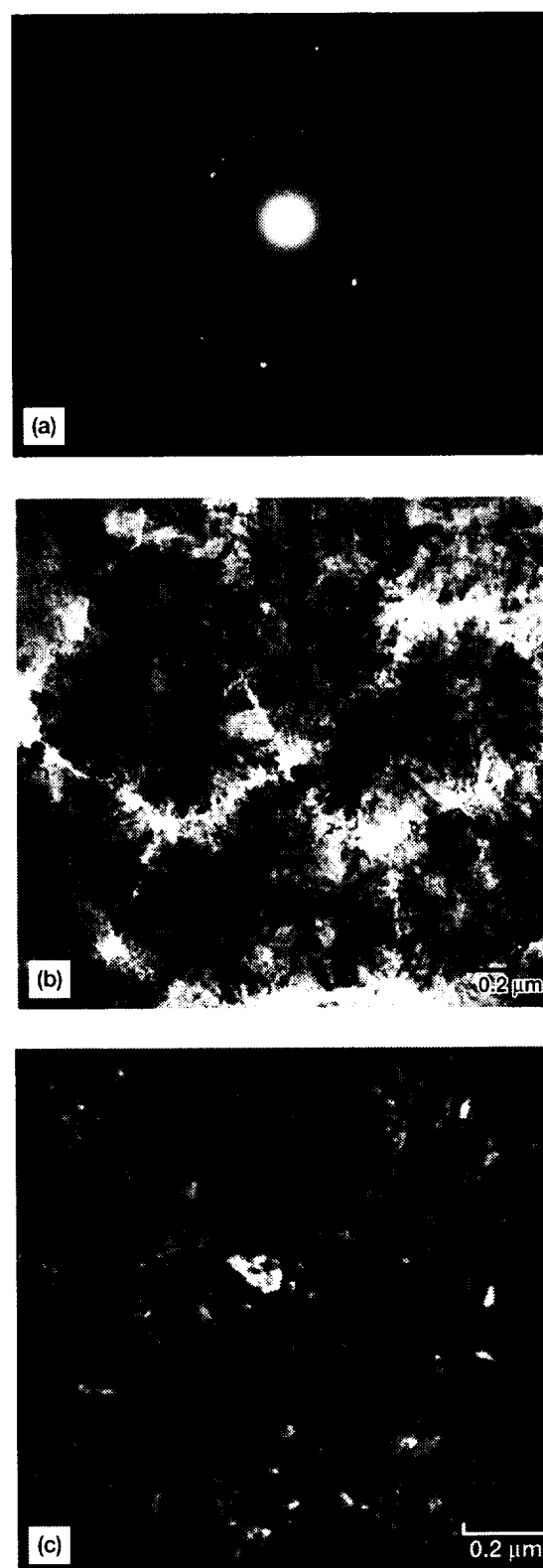


Figure 5.—Photographs of free-standing diamond films. (a) Selected-area diffraction pattern. (b) Bright-field TEM. (c) Dark-field TEM.

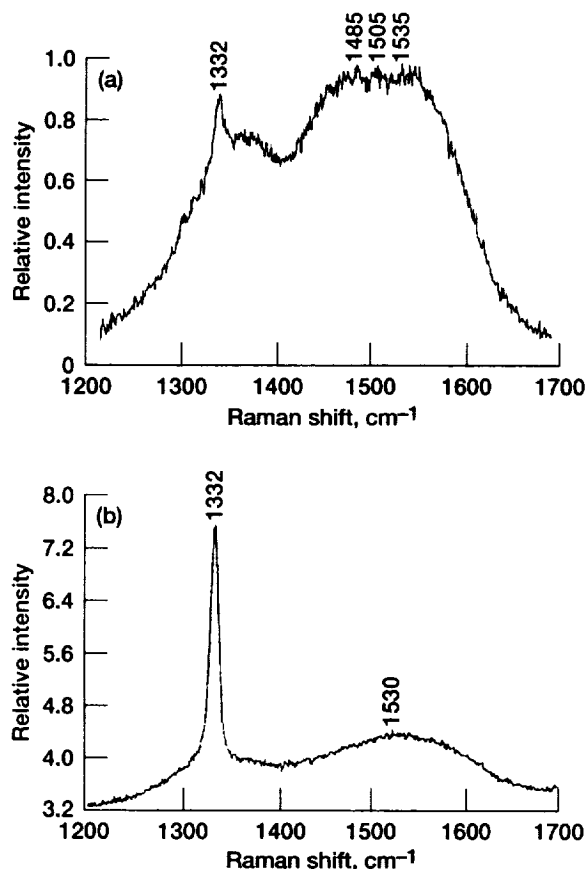


Figure 6.—Raman spectra of diamond films. (a) Fine-grain (20 to 100 nm) diamond film on {100} silicon substrate. (b) Medium-grain (1100 nm) diamond film on {100} silicon substrate.

## 2.4 Rutherford Backscattering Spectroscopy and Elastic Recoil Spectroscopy

Rutherford backscattering is a nondestructive, quantitative depth profiling of thin-film compositions and structures, crystallinity, dopants, and impurities [8]. Elastic recoil spectroscopy (hydrogen forward scattering or proton recoil detection) is the simplest ion beam technique for hydrogen profiling and determines hydrogen concentrations in thin films. In combination with RBS analysis of the same sample ERS provides concentration profiles and complete compositional analysis of the near-surface regions of the sample material.

Figures 7 and 8 [4] present RBS and ERS spectra, respectively, of a fine-grain CVD diamond film. Besides carbon from the diamond film and silicon from the silicon substrate, no other elements are observed in the RBS spectrum. From both spectra it was estimated that the fine-grain diamond film consisted of 97.5 at.% carbon and 2.5 at.% hydrogen. (In contrast, the medium-grain diamond films contained less than 1 at.% hydrogen [5].)

It was also demonstrated that both carbon and hydrogen are uniformly distributed in the fine-grain film from the top of the surface to the silicon substrate.

RBS analytical results can also be used to determine diamond film thickness. Figure 7 presents a simulated RBS spectrum of the diamond film containing a carbon-to-hydrogen ratio (C/H) of 97.5/2.5 obtained by using the RUMP computer code [13]. In the computer program the film thickness of the diamond film is taken as a variable. This thickness was obtained from the close match between the observed and simulated RBS, as shown in Fig. 7, and is 1.5  $\mu\text{m}$  at the center of the substrate. The deposition rate was estimated to be 0.14  $\mu\text{m/hr}$ .

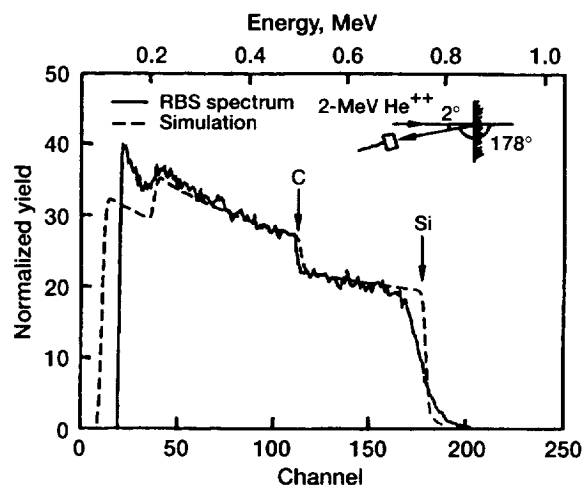


Figure 7.—Rutherford backscattering spectrum of fine-grain diamond film on silicon substrate. (Simulation curve was calculated by using the computer code RUMP.)

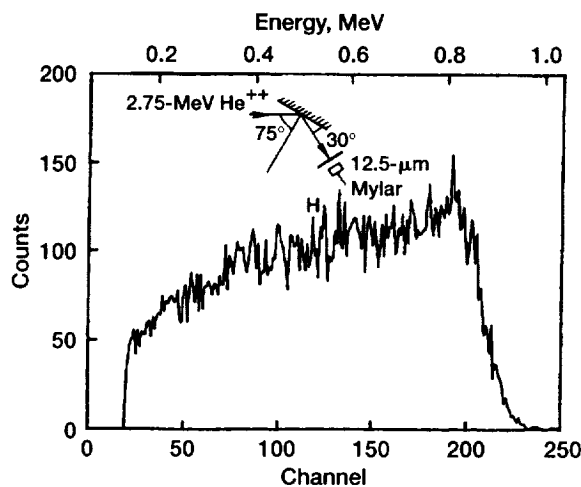


Figure 8.—Hydrogen forward-scattering spectrum (proton recoil analysis) of fine-grain diamond film on silicon substrate.



## 2.5 Tribological Examination

The classical Bowden and Tabor model for sliding friction of materials in contact, in its simple form, assumes that the friction force arises from two contributing sources [14]. First, an adhesion force is developed at the real area of contact between the surfaces, arising from the attractive forces between the contacting surfaces. Second, a deformation force is needed to plow or cut the asperities of the harder surface through the softer. The resultant friction force is the sum of the two contributing sources: friction due to adhesion and friction due to deformation and/or fracture. When a smooth flat is brought into contact with a smooth spherical surface of the same or softer material, the plowing or cutting contribution in friction can be neglected. In this situation, as is well known, diamond is one of the slipperiest materials and is similar to polytetrafluoroethylene in air. The coefficient of friction is between 0.01 and 0.05.

When the fine-, medium-, and coarse-grain diamond films were brought into contact with a natural diamond pin in reciprocating sliding motion in air, the coefficients of friction varied as the pin traveled back and forth (reciprocating motion), retracing its tracks on the diamond films (Fig. 9). In humid air at a relative humidity of 40% abrasion (plowing) occurred and dominated the friction and wear behavior. The bulk natural diamond pin tended to dig into the surface of the diamond films during sliding and produce a wear track (groove). SEM observations of the diamond films indicated that small fragments chipped off their surfaces. When abrasive interactions between the diamond pin surface and the initially sharp tips of

asperities on the diamond film surfaces were strong, the friction was high (point A in Fig. 9). The surface roughness of diamond films can have an appreciable influence on the initial friction of diamond films: the greater the initial surface roughness, the higher the initial coefficient of friction. Similar frictional results have also been found by other workers on single-crystal diamonds [15] and on diamond coatings [16].

As sliding continued and the pin passed repeatedly over the same track, the coefficient of friction was appreciably affected by the wear (i.e., a blunting of the tips of the asperities) on the diamond films (Fig. 9). When repeated sliding produced a smooth groove or a groove with blunted asperities on the diamond surface (Fig. 10), the coefficient of friction was low ( $<0.05$ ). The initial surface roughness effect became negligible. Therefore, the equilibrium coefficient of friction was independent of the initial surface roughness of the diamond film.

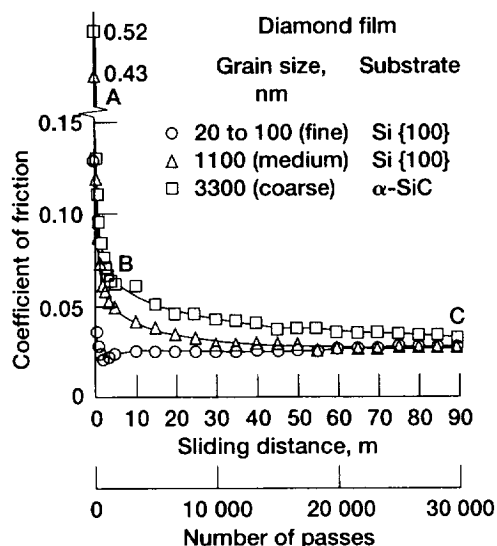


Figure 9.—Coefficient of friction as function of number of passes of bulk diamond pin in contact with fine-, medium-, and coarse-grain diamond films in humid air (approx. 40% relative humidity).

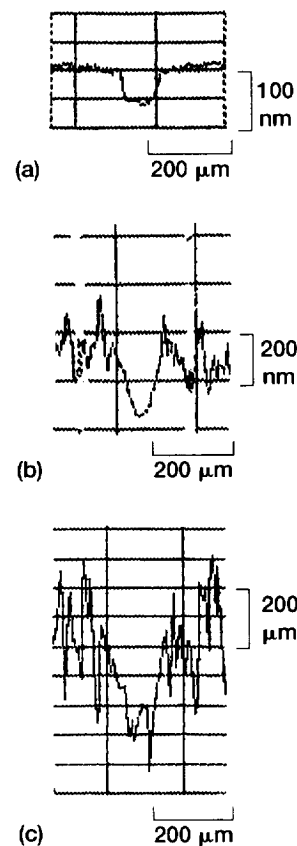


Figure 10.—Wear tracks (grooves) on diamond films after 30 000 passes of bulk diamond pin in dry nitrogen. (a) Fine-grain (20 to 100 nm) diamond film; rms surface roughness, 15 nm. (b) Medium-grain (1100 nm) diamond film; rms surface roughness, 63 nm. (c) Coarse-grain (3300 nm) diamond film; rms surface roughness, 160 nm.

### 3.0 Concluding Remarks

To achieve satisfactory surface and bulk properties of coatings and films, it is necessary to optimize deposition parameters. These parameters must not only give the appropriate initial level of surface and bulk properties but must also provide the durability of films and coatings. To understand the benefits provided by deposition parameters, and ultimately to provide better deposition parameters, it is necessary to study the physical, chemical, and structural changes of films and coatings produced by these deposition parameters.

The use of analytical techniques including tribological examination was highlighted in the important case study of microwave-plasma-assisted, chemical-vapor-deposited diamond films for tribological applications. Each diagnostic technique provides unique information. It should be possible to coordinate the different pieces of information provided by these diagnostic techniques into a coherent self-consistent description of the surface properties. These methodologies have contributed significantly to the understanding of the characteristics and properties of diamond films and their surfaces.

A combination of surface diagnostic techniques was successfully applied to provide correlation between deposition parameters and resultant diamond film composition, crystallinity, grain size, and surface roughness.

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